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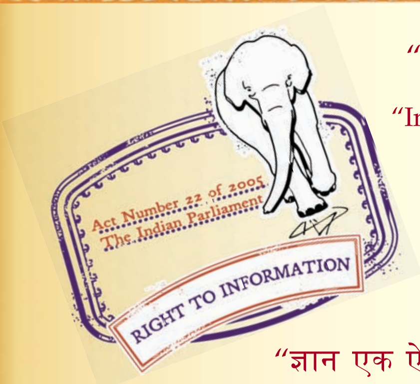

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IS : 6361 - 1971
(Reaffirmed 2003)

Indian Standard

METHODS OF COLORIMETRIC DETERMINATION OF PHOSPHORUS

(Second Reprint MAY 1988)

UDC 546.18:543.43

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2 *April* 1972

Indian Standard
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METHODS OF COLORIMETRIC DETERMINATION OF PHOSPHORUS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 12 November 1971, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The determination of traces of phosphate is one of the most commonly performed determinations in colorimetric analysis. In the colorimetric determination of phosphorus it is generally required that the phosphorus shall be present as orthophosphate. Suitable treatments should be given prior to determination to convert them as orthophosphates.

0.3 The colorimetric methods for determination of phosphorus are of choice these days and increasingly employed in analytical works for determination of micro-quantities. Colorimetric methods are more accurate, sensitive and quicker compared to volumetric methods.

0.4 This standard prescribes methods of determination of phosphorous in traces by colorimetry. The volumetric determination of phosphorous is covered by IS : 5305-1969*.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard prescribes the colorimetric methods for the determination of phosphorus.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960‡) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Methods for volumetric determination of phosphorus.

†Rules of rounding off numerical values (*revised*).

‡Specification for water, distilled quality (*revised*).

3. METHOD BASED ON THE REDUCTION WITH STANNOUS CHLORIDE

3.0 Outline of the Method — A blue colour is produced by the reduction of phosphomolybdic acid with freshly prepared stannous chloride solution.

3.1 Apparatus

3.1.1 *Separating Funnels* — of 200 ml capacity.

3.1.2 *Nessler Cylinders* — of 50 ml capacity. (*see* IS : 4161-1967*).

3.1.3 p*H Meter* — glass electrode type.

3.2 Reagents

3.2.1 *Ammonium Molybdate Solution* — Dissolve 10 g of ammonium molybdate in 100 ml of water. When cool, add the solution to 300 ml of 1 : 1 sulphuric acid. Keep the reagents in a glass bottle, protected from light.

3.2.2 *Stannous Chloride Solution* — Warm 0·1 g of tin foil in 2 ml of hydrochloric acid and a drop of 4 percent (*w/v*) copper sulphate solution in a test tube until no more tin dissolves, cool and dilute to 10 ml with water. The reagent shall be prepared freshly for each determination.

3.2.3 *Standard Phosphate Solution* — Dissolve 3·77 g of sodium hydrogen phosphate ($Na_2HPO_4.12H_2O$) in water and dilute to 1 000 ml. One millilitre of the solution contains 1·0 mg of phosphate (as PO_4). It may be diluted suitably to contain 10, 25, 50 or 100 µg of phosphate per millilitre.

3.2.4 *Ether* — Conforming to IS : 336-1964†.

3.2.5 *Hydrochloric Acid* — conforming to IS : 265-1962‡.

3.3 Procedure

3.3.1 Prepare the solution as prescribed in the individual material specifications. Take a convenient aliquot of the prepared solution, so as to contain 0·025 mg of phosphorus (as P_2O_5). Transfer the solution to a platinum dish and dilute to 50 ml. Digest on a steam-bath for 20 minutes. Cool, adjust the *p*H to about 4 and dilute to 75 ml. Add 1 ml of ammonium molybdate reagent and mix well. When it is dissolved adjust the *p*H to 2 by adding dilute hydrochloric acid. Check the *p*H using *p*H meter (glass electrode). Heat to boiling, cool to room temperature, add 10 ml of concentrated hydrochloric acid and dilute to 100 ml with water. Transfer the solution to a separating funnel, add 35 ml of ether, shake vigorously and

*Specification for Nessler cylinders.

†Specification for ether (*revised*).

‡Specification for hydrochloric acid (*revised*).

allow to separate. Draw off the aqueous phase which may contain silicates and discard. Wash the ether phase with 10 ml of hydrochloric acid and allow to separate. Drain off and discard this aqueous phase. Drain the ether layer quantitatively to a Nessler cylinder and develop the colour by adding 0·15 ml of stannous chloride reagent. Mix well and make up to mark with ether.

3.3.2 Transfer several aliquots of the standard phosphate solution to separating funnels and develop the colour following the steps described in **3.3.1**.

3.3.3 Compare the colour with those produced with the standard phosphate solution. Note the volume of the standard phosphate solution with which the colour of the test solution matches closely.

3.3.4 *Calculation*

$$\text{Phosphate (as } PO_4 \text{), percent by mass} = \frac{100 \times f \times V}{M}$$

where

f = mass in g of phosphorus (as PO_4) equivalent to 1 ml of standard phosphate solution,

V = volume in ml of standard phosphate solution matching closely with colour of the test solution, and

M = mass in g of the material in the aliquot used for test.

NOTE — The colour is produced only in presence of orthophosphates. Meta and pyro phosphate should be completely hydrolyzed before determination. Free acids and alkalis which depress the colour development should be neutralized. Organic acids like citric, tartaricand oxalic inhibit the colour formation. If organic matter is present in appreciable amounts, it should be removed. Ferric iron exceeding 1 ppm should be reduced to ferrous iron as well as arsinate to assenite.

4. INSTRUMENTAL METHOD

4.0 General

4.0.1 *Outline of the Method* — The sample is neutralized and reacted with ammonium molybdate and stannous chloride. The blue colour obtained is matched against that produced with standard phosphate solution.

4.1 Apparatus

4.1.1 *Spectrophotometer* — for use at 730 nm.

4.1.2 *Filter Photometer* — Providing a light path of 1 to 10 cm and equipped with a red filter having a mximum transmittance around 730 nm.

4.1.3 p*H Meter* — of glass electrode type.

4.1.4 *Burette* — to an accuracy of 0·1 ml.

4.1.5 *Separating Funnel* — of 200 ml capacity.

4.2 Reagents

4.2.1 *Isobutanol*

4.2.2 *Nitric Acid* — approximately 8 N.

4.2.3 *Sulphuric Acid* — approximately 10 N.

4.2.4 *Sulphuric Acid* — approximately 0·5 N.

4.2.5 *Ammonium Acetate Solution* — 50 percent (*w/v*).

4.2.6 *Ferric Sulphate Solution* — Dissolve 0·5 g of ferric sulphate [$Fe_2(SO_4)_3 \cdot 9H_2O$] in 50 ml of water containing 2 ml of perchloric acid and make up the volume to 100 ml. One millilitre of this solution contains 0·001 g of iron approximately.

4.2.7 *Ferrous Ammonium Sulphate Solution* — Dissolve 0·5 g of ferrous ammonium sulphate [$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$] with 50 ml of water containing 0·5 ml of perchloric acid and make up the volume to 100 ml. One millilitre of this solution contains 0·007 g approximately of iron. Prepare the solution fresh before use.

4.2.8 *Ammonium Molybdate Solution* — 25 g per 1 000 ml. Dissolve 5 g of ammonium molybdate [$(NH_4)_6 MoO_4 \cdot 4H_2O$] in water at 60°C. Cool and make up the volume to 100 ml. Add to the solution 100 ml of sulphuric acid (**4.2.4**) and mix.

4.2.9 *Washing Solution* — Saturate at room temperature about 500 ml of sulphuric acid with isobutanol.

4.2.10 *Stannous Chloride Solution* — containing 2·38 g per litre. Dissolve 1·19 g of stannous chloride ($Sn\,Cl_2 \cdot 2H_2O$) in 85 ml of hydrochloric acid (d 1·19). Complete the volume to 500 ml. Preserve the solution in amber coloured bottles. Prepare the solution fresh before use.

4.2.11 *Standard Phosphorus Solution* — 0·400 g per litre of P_2O_5. Weigh accurately 0·766 8 g of potassium dihydrogen phosphate (KH_2PO_4), dried before hand over sulphuric acid, transfer quantitatively to a 1 000 ml volumetric flask, make up to the mark with the water and mix. One millilitre of this solution contains 0·4 mg of P_2O_5.

4.2.11.1 Pipette out 25 ml of the solution (**4.2.11**) in a 1 000 ml volumetric flask and make up the solution to mark and mix. One millilitre of this solution contains 0·01 mg of P_2O_5.

4.2.11.2 Pipette out 25 ml of the solution (**4.2.11.1**) into a 250 ml volumetric flask and make up to mark and mix. One millilitre of this solution contains 1 µg of P_2O_5.

4.3 Procedure

4.3.1 *Preparation of the Main Test Solution* — Prepare the solution of the material as prescribed in individual material specification.

4.3.2 *Test Specimen* — Take a volume of the test solution containing 0·015 to 0·025 mg of P_2O_5.

4.3.3 *Blank Test* — Carry out simultaneous with the actual test a blank also using the same quantities of all the reagents those are employed for the test sample under same conditions.

4.3.4 *Establishment of Calibration Curve*

4.3.4.1 *Preparation of reference solution — Relating to spectrophotometric measurements carried out with an optical range of 1 cm.* Introduce in a series of 6 separating funnels, the volumes of the standard phosphorous solution as shown below, measured from a burette:

Standard Phosphorus Solution (**4.2.11.2**)	*Corresponding P_2O_5 Content*
ml	mg
0*	0
5·0	0·005
10·0	0·010
15·0	0·015
20·0	0·020
25·0	0·025

Treat each of these solutions as follows:

Add to these separating funnels the necessary amount of water to bring the volume to 60 ml, 1 ml of ferric sulphate and 1 ml of ferrous ammonium sulphate solutions and mix well.

4.3.4.2 *Formation of oxidized phosphomolybdic complex and extraction of the reduced complex* — Add to these separating funnels 3 ml of sulphuric acid (**4.2.4**) and then 7·5 ml of ammonium molybdate solution. Mix and let stand for 10 minutes. Add 25 ml of isobutanol and shake vigorously for 1 minute. Decant, separate the aqueous phase and reject it. Add to the organic phase 30 ml of washing solution, shake for 1 minute. Separate the aqueous phase and reject it. Repeat the washings once. Transfer for organic phase to a 25 ml volumetric flask dried beforehand. Wash the separating funnel with 1 to 2 ml of isobutanol and receive the washings also in the same volumetric flask. Then, complete the volume to mark with same isobutanol. Mix and let stand for at least 10 minutes away from light. Add about 2 ml of stannous chloride solution.

*Compensation solution.

4.3.4.3 *Spectrophotometric measurement* — After 10 minutes, but before 1 hour, carry out the spectrophotometric measurements at a wavelength of about 730 nm, the instrument being corrected before for zero absorbance with respect to the compensation solution.

4.3.4.4 *Plotting of the Calibration Curve* — Plot the graph taking the values in mg of P_2O_5 of *X*-axis and the values for absorbance on *Y*-axis.

4.3.4.5 *Determination* — Transfer the test portion to a separating funnel (adjust the *p*H by a glass-electrode *p*H meter to 2 ± 0·1 using small amounts of ammonium acetate). Then bring the volume to about 60 ml and add about 1 ml of ferric sulphate solution, 1 ml of ferrous ammonium sulphate solution and mix.

4.3.4.6 *Formation of the complex and extraction of the reduced complex* — Proceed as prescribed in **4.3.4.2**.

4.3.4.7 *Measurement of spectrophotometry* — Measure the spectrophotometry of the organic phases resulting from the test and blank test solutions as prescribed in **4.3.4.3** after adjusting the instrument for zero absorbance with respect to isobutanol.

4.3.5 *Calculation* — By means of the calibration curve determine the quantity of P_2O_5 corresponding to the values of absorbance.

$$\text{Phosphorous (as } P_2O_5 \text{), percent by weight} = \frac{M_1 - M_2}{M_3} \times V \times 100$$

where

M_1 = mass in g of P_2O_5 found in the test solution,

M_2 = mass in g of P_2O_5 found in the blank solution,

V = the ratio of the volume of the main solution(s) to that of test portion, and

M_3 = mass in g of the material in test solution prepared from the main solution (**4.3**).

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